APPLICATION FOR UNITED STATES LETTERS PATENT

CERAMIC TO NOBLE METAL BRAZE AND METHOD OF MANUFACTURE

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CERAMIC TO NOBLE METAL BRAZE AND METHOD OF MANUFACTURE

FIELD OF THE INVENTION

This invention relates to a hermetically sealed ceramic to metal bond joint and method of producing the bond.

BACKGROUND OF THE INVENTION

Known methods of bonding a ceramic to a metal involve the use of interlayer materials which either melt at the bonding temperature, such as a braze, or which involve special coating processes for the material surfaces to be bonded, such as pre-coating the surfaces with an activating material. In some methods of bonding, an interlayer material having a composition that approximates the composition of the initial metal bonding surface is utilized, such as disclosed by Lasater (U.S. Patent No. 6,221,513). Lasater discloses a method for forming a hermetically sealed bond for use in implantable medical devices. Hill (U.S. Patent No. 3,594,895) discloses another approach to forming a ceramic to metal seal. Fey, et al. (U.S. Patent No. 6,521,350) discloses the use of pure nickel or nickel alloy to bond titanium to a ceramic, such as alumina or zirconia.

Cusano (U.S. Patent No. 3,994,430) discloses a method of directly bonding metal to ceramic substrates where a very thin layer of an interlayer material is placed between the metal and the ceramic to be bonded. The system is heated in an inert atmosphere to a temperature between the eutectic temperature of the interlayer material and the melting point of the metal. Cusano focuses on bonding copper to a ceramic substrate, such as alumina or beryllia.

In one application, it is desired to have a platinum eyelet attached to a titanium alloy end cap that is in turn bonded to a hollow ceramic tube, which is implantable in living tissue as, for example, a microstimulator or microsensor. The BION of Advanced Bionics Corporation is one such device. The inventors

have demonstrated that welding creates cracks at the platinum eyelet and the titanium alloy end cap weld joint. Low tensile strength has been measured in these welded joints. Continuous welding yielded an average strength of 4.5 lbf; double pass continuous welding yielded 4.5 lbf; and single shot welding yielded 16.2 lbf.

A hermetic, strong end cap that is bonded to a ceramic is needed, especially for implantable components.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 illustrates the side view of the component assembly with the interlayer material as a foil between the ceramic and metal parts.

FIG. 2 schematically depicts the bonding steps of the present invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

FIG. 1 shows component assembly 2 having metal part 4, ceramic part 6, and interlayer material 8. Component assembly 2 is heated to a specific process temperature, that is below the melting point of metal part 4, for a specific period of time, at a pressure that is created by force 10 and that is exerted to place interlayer material 8 in intimate contact with the metal and ceramic parts.

Interlayer material 8 is a foil having a thickness of 0.003 of an inch or less. The interlayer material 8 is preferably a clad foil comprised of at least one layer of nickel and one layer of titanium. Layering allows liquidus formation in the foil at a lower temperature than with an alloy of equivalent composition. In an alternative embodiment, the interlayer material 8 may be comprised of foil layers as a laminate of nickel and titanium that are in intimate contact, but that are not clad bonded to one another. In a further alternative embodiment, the interlayer material 8 is comprised of multiple foil layers of nickel and titanium. For simplicity, clad foil and layers are referred to as laminates herein.

Interlayer material 8 is selected from the group of materials that are compatible with the ceramic chosen for ceramic part 6 in that they wet the surface during the bonding process and chemically react with the ceramic part 6 thereby creating a strong bond joint during processing. Interlayer material 8 also is selected from the group of materials that are compatible with the metal chosen for metal part 4. Interlayer material 8 forms a bond with a metal part 4 by virtue of developing alloys, intermetallics or solid solution at the bonding temperature and pressure utilized during processing. The reaction products formed during processing is composed of the metal selected for metal part 4 and the metals selected for interlayer material 8. The group of interlayer materials includes titanium-nickel. In a preferred embodiment, interlayer material 8 is titanium-nickel foil having at least 50.0% and less than 67.0% of titanium with a thickness of approximately 0.002 inches.

Metal part 4 is a biocompatible material selected from the group of noble metals such as platinum, iridium, palladium, ruthenium, rhodium, or alloys thereof. A preferred embodiment is 90% by weight platinum and 10% iridium.

Alternative embodiments include, but are not limited to, pure platinum, pure iridium, alloys of platinum and rhodium. Ceramic part 6 may be alumina, titania, zirconia, stabilized-zirconia, partially-stabilized zirconia, tetragonal zirconia polycrystal, tetragonal zirconia polycrystal, magnesia-stabilized zirconia, ceria-stabilized zirconia, yttria-stabilized zirconia, and calcia-stabilized zirconia, and in a preferred embodiment ceramic part 6 is tetragonal zirconia polycrystal. In alternative embodiments, rather than using interlayer material 8 as a foil, interlayer material 8 may be a thin coating that is applied to either the metal part 4 or ceramic part 6 surface to be bonded by any of a variety of chemical processes such as electroless plating and electroplating, or by any of a variety of thermal processes such as sputtering, evaporating, or ion beam enhanced deposition. In a preferred embodiment, the coating may be layers of nickel and titanium. Interlayer material 8 may also be applied as a thin coating of metallic beads or metallic powder. It is preferred that the beads or powder be comprised of separate beads of essentially pure titanium and pure nickel, to lower the liquidus formation temperature during brazing.

The process steps that are employed to create assembly 2 with a strong bond between metal part 4 and ceramic part 6 are schematically represented in **FIG. 2**. First, the surfaces to be bonded are prepared in step 20 by machining to assure that they will intimately conform to each other during bonding. The surfaces are smoothed and cleaned.

In step 22, component assembly 2 is prepared with interlayer material 8 between metal part 4 and ceramic part 6. In step 24, force 10 is applied to compress interlayer material 8 between metal part 4 and ceramic part 6. Force 10 is sufficient to create intimate contact between the parts. Force 10 is applied to assure that a strong bond is formed between metal part 4 and ceramic part 6 thus creating a hermetic seal between the two parts.

In step 26 the assembly to be heat processed is placed in a furnace in a non-reactive environment, which is preferably vacuum, but which can be argon atmosphere in an alternative embodiment. A vacuum is applied before the furnace is heated to the processing temperature in step 28. A preliminary

holding temperature may be used to allow the thermal mass of the parts to achieve equilibrium before proceeding with heating. The process temperature is lower than the melting point of metal part 4, but greater than the temperature of the eutectic formed between the metals of the interlayer material 8. In a preferred embodiment, the vacuum is 10^{-5} to 10^{-7} torr, to assure that the interlayer material 8 and metal part 4 do not oxidize. Component assembly 2 is held at the selected temperature, which is typically between approximately 960° and 1080°C, for approximately 1 to 60 minutes, while force 10 continues to be exerted on interlayer material 8. The exact time, temperature and pressure are variable to achieve a hermetic and strong bond of metal part 4 with ceramic part 6. For example, in a preferred embodiment, a tetragonal zirconia polycrystal, TZP, part is bonded to a platinum part in vacuum of 10^{-5} torr at approximately 1060°C for 10 minutes with a pressure of approximately 20 to 800 psi on a 50% by weight nickel and 50% titanium foil of approximately 0.002 inches thickness. Alternately, the foil is 67% titanium and 33% nickel, such as Tini-67®. One source of such materials is Morgan Advanced Ceramics. In some cases the foil may be an alloy, although a laminate material has been demonstrated to effect a strong bond under the conditions demonstrated herein.

The furnace is cooled and component assembly 2 is cooled to room temperature in step 30. In optional step 32, component assembly 2 is cleaned by being placed in a bath, after thermal processing is complete, to assure removal of all nickel and nickel salts. This bath is preferably an acid bath that etches the exposed surfaces of component assembly 2. In a preferred embodiment, the bath is nitric acid. Removal of nickel and nickel salts in the bath etch insures that component assembly 2 is biocompatible. Nickel and nickel salts can be detrimental to living animal tissue. In the preferred embodiment, however, all of the nickel that is introduced as interlayer material 8 is combined with the titanium or platinum and is tied up to be unavailable as free nickel or as a nickel salt.

Component assembly 2 is biocompatible after proper bonding and processing. Metal part 4, ceramic part 6, and interlayer material 8 are selected

so as to be compatible with the environment in a living body. Hence, metal part 4 is preferably a platinum alloy and ceramic part 6 is preferably TZP.

In a preferred embodiment, component assembly 2 is either an electrical sensor or an electrical stimulator that is implanted in a human body, although it could equally well be implanted in any animal. It must survive long periods in the hostile environment of a living body, which is basically a warm saline solution. In a preferred embodiment, component assembly 2 is either a sensor or stimulator comprised of a hollow ceramic tube that contains various electronic components that is bonded to a metal electrode end. The component assembly must be watertight; hence, the bond is hermetic, resisting salt-water intrusion as well as growth of living tissue into the metal-to-ceramic bond joint.

Further, component assembly 2 does not corrode while implanted in the body. The materials are chosen such that post-bonding they are not susceptible to corrosion either individually or in the as-bonded state. Component assembly 2 resists electrolytic corrosion as well as crevice corrosion, because resistant materials are selected for component assembly 2.

Obviously, many modifications and variations of the present invention are possible in light of the above teachings. It is therefore to be understood that, within the scope of the appended claims, the invention may be practiced otherwise than as specifically described.